

What is claimed is:

1                   1.     A method of making a hexammine cobaltic salt, the method  
2 comprising:

3                   (a) providing a reaction vessel containing an aqueous solution of at least  
4 one ammonia source selected from the group consisting of ammonium hydroxide,  
5 ammonia-containing gas and combinations thereof;

6                   (b) introducing a solution of  $\text{Co}(\text{X})_2$  and a solution of  $\text{NH}_4\text{X}$ , wherein  
7 X is at least one selected from the group consisting of chloride, bromide, perchlorate,  
8 and nitrate, into the reaction vessel to form reaction vessel contents comprising a first  
9 product precursor;

10                  (c) introducing an oxygen-containing gaseous oxidizer to the reaction  
11 vessel contents of (b) to form an at least partially oxidized second product precursor;

12                  (d) heating the at least partially oxidized second product precursor to  
13 form a third product precursor;

14                  (e) adding a surface active catalyst to the third product precursor;

15                  (f) continue introducing an oxygen-containing gaseous oxidizer to the  
16 catalyst-containing reaction mixture of (e) while maintaining the temperature at a  
17 temperature selected to result in product formation of a desired particle size; and

18                  (g) cooling the mixture from (f) and recovering the hexammine cobaltic  
19 salt therefrom as an admixture with the surface active catalyst.

1                    2.     The method of claim 1 wherein X is nitrate and the hexammine  
2 cobaltic salt is hexammine cobaltic nitrate.

1                    3.     The method of claim 2 wherein the first product precursor  
2 comprises  $\text{Co}(\text{NH}_3)_5(\text{H}_2\text{O})(\text{NO}_3)_2$ .

1                    4.     The method of claim 2 wherein the at least partially oxidized  
2 second product precursor comprises  $\mu$ -peroxobis [pentamminecobalt].

1                    5.     The method of claim 2 wherein the third product precursor  
2 comprises pentammine-aqua cobaltic nitrate.

1                    6.     The method of claim 2 wherein the at least partially oxidized  
2 second product precursor is heated in (d) to a temperature in a range of about 95°F to  
3 about 120°F.

1                    7.     The method of claim 2 wherein in (f) the temperature is  
2 maintained in a range of about 95°F to about 120°F.

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1                    8.     The method of claim 1 wherein the providing of the aqueous  
2     solution of an ammonia source in the reaction vessel in (a) comprises adding the  
3     ammonia source to the reaction vessel in an amount sufficient to provide about 5.5 to  
4     about 6.5 equivalents of ammonia to cobalt.

1                    9.     The method of claim 1 wherein the solution of  $\text{Co(X)}_2$  and  $\text{NH}_4\text{X}$   
2     comprises water,  $\text{Co(NO}_3)_2$  and ammonium nitrate.

1                    10.    The method of claim 1 wherein the surface active catalyst  
2     comprises activated carbon.

1                    11.    The method of claim 10 wherein the activated carbon surface  
2     active catalyst is added in an amount sufficient to provide no more than about 1.5 %  
3     carbon in the hexammine cobaltic salt.

1                    12.    The method of claim 11 wherein the activated carbon surface  
2     active catalyst is added in an amount sufficient to provide no more than about 0.5 %  
3     carbon in the hexammine cobaltic salt.

1                   13.    The method of claim 12 wherein the activated carbon surface  
2    active catalyst is added in an amount sufficient to provide about 0.2-0.3 % carbon in  
3    the hexammine cobaltic salt.

1                   14.    The method of claim 1 wherein the oxygen-containing gaseous  
2    oxidizer is introduced to the heated combination in (c) to form an at least partially  
3    oxidized reaction mixture having an absorbance @ 505 nm of at least about 1.5 AU.

1                   15.    The method of claim 1 wherein the hexammine cobaltic salt  
2    recovered in (f) is of a particle size in the range of about 35 microns to about 60  
3    microns.

1                   16.    The method of claim 1 wherein the reaction vessel contents of (b)  
2    are heated prior to the initiation of the introduction of the oxygen-containing gaseous  
3    oxidizer.

1                   17.    The method of claim 1 wherein the introduction of the solution  
2    of  $\text{Co(X)}_2$  and the solution of  $\text{NH}_4\text{X}$  comprises introducing a solution containing both  
3     $\text{Co(X)}_2$  and  $\text{NH}_4\text{X}$ .

18. A method of making a hexammine cobaltic nitrate having a particle size in the range of about 35 microns to about 60 microns, the method comprising:

(a) providing a reaction vessel containing an aqueous solution of at least one ammonia source selected from the group consisting of ammonium hydroxide, an ammonia-containing gas and combinations thereof;

(b) introducing a solution of  $\text{Co}(\text{NO}_3)_2$  and  $\text{NH}_4\text{NO}_3$  into the reaction vessel to form reaction vessel contents comprising a first product precursor;

(c) heating the first product precursor to a temperature in the range of 95°F to 120°F;

(d) introducing an oxygen-containing gaseous oxidizer to the heated first product precursor in (c) to form an at least partially oxidized reaction mixture having an absorbance @ 505 nm of at least about 1.5 AU;

(e) adding a surface active catalyst of activated carbon to the reaction mixture of (d);

(f) continue introducing an oxygen-containing gaseous oxidizer to the catalyst-containing reaction mixture of (e) while maintaining the temperature within a range of about 95°F to about 120°F and which temperature is selected to result in to result in the hexammine cobaltic nitrate having a desired particle size; and

(g) cooling the mixture from (f) and recovering the hexammine cobaltic nitrate therefrom as an admixture with the surface active catalyst.

19. The method of claim 18 wherein the providing of the aqueous solution of an ammonia source in the reaction vessel in (a) comprises adding the ammonia source to the reaction vessel in an amount sufficient to provide about 5.5 to about 6.5 equivalents of ammonia to cobalt.

20. The method of claim 18 wherein the activated carbon surface active catalyst is added in an amount sufficient to provide no more than about 1.5 % carbon in the hexammine cobaltic salt.

21. The method of claim 18 wherein in (d), the oxygen-containing gaseous oxidizer is introduced to the heated first product precursor in (c) to form an at least partially oxidized reaction mixture having an absorbance @ 505 nm of about 2.5 AU.

22. A method of making a hexammine cobaltic nitrate having a selected particle size, the method comprising:

(a) providing a reaction vessel containing an aqueous solution of at least one ammonia source selected from the group consisting of ammonium hydroxide, ammonia-containing gas and combinations thereof;

(b) introducing a solution of  $\text{Co}(\text{NO}_3)_2$  and  $\text{NH}_4\text{NO}_3$  into the reaction vessel to form reaction vessel contents comprising  $\text{Co}(\text{NH}_3)_5(\text{H}_2\text{O})(\text{NO}_3)_2$ ;

(c) introducing an oxygen-containing gaseous oxidizer to the reaction vessel contents of (b) to form  $\mu$ -peroxobis [pentamminecobalt];

(d) heating the  $\mu$ -peroxobis [pentamminecobalt] to form pentammine-aqua cobaltic nitrate;

(e) adding a surface active catalyst of activated carbon to the pentammine-aqua cobaltic nitrate;

(f) continue introducing an oxygen-containing gaseous oxidizer to the catalyst-containing reaction mixture of (e) while maintaining the temperature at a selected temperature in the range of about 95°F to about 120°F to result in the hexammine cobaltic nitrate having a selected particle size in the range of about 35 microns to about 60 microns; and

(g) cooling the mixture from (f) and recovering the hexammine cobaltic nitrate therefrom as an admixture with the surface active catalyst.

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1                   23.    The method of claim 22 wherein the providing of the aqueous  
2    solution of the at least one ammonia source in the reaction vessel in (a) comprises  
3    adding the ammonia source to the reaction vessel in an amount sufficient to provide  
4    about 5.5 to about 6.5 equivalents of ammonia to cobalt.

1                   24.    The method of claim 22 wherein the activated carbon surface  
2    active catalyst is added in an amount sufficient to provide no more than about 1.5 %  
3    carbon in the hexammine cobaltic salt.

1                   25.    The method of claim 22 wherein the oxygen-containing gaseous  
2    oxidizer is introduced to the heated combination in (c) to form an at least partially  
3    oxidized reaction mixture having an absorbance @ 505 nm of at least about 1.5 AU.

1                   26.    The method of claim 22 wherein the reaction vessel contents of  
2    (b) are heated prior to the initiation of the introduction of the oxygen-containing  
3    gaseous oxidizer.